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ULTRAHIGH PRESSURE RESEARCH — ITS PROGRESS AND PROMISE

MONOGRAPH SERIES

by

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U. S. ARMY MATERIALS RESEARCH AGENCY

ULTRAHIGH PRESSURE RESEARCH - ITS PROGRESS AND PROMISE

ABSTRACT

Ultrahigh pressure, how it is produced, the kind of machinery required, the "massive support" principle and its evolution, together with the effect of extreme pressure on the basic structure of some materials are discussed. A bibliography is attached.

INTRODUCTION

One of the major concerns of the materials scientist is the relation between the basic structure of an element or an alloy and the properties it exhibits. Changes in basic structure can bring with them favorable alterations in both the physical and mechanical properties of metals, alloys, and nonmetallic compounds. By applying extreme pressures to some materials we are able to change their lattice structure or to actually alter the natural alignment of the atoms within their crystals. When this happens in such a manner that the new alignment does not revert to its former undisturbed condition, we have an entirely different, in fact, a new material before us. A known and rather striking example of a transformation of this kind is the conversion, under very high static pressure and temperature, of graphite into diamond. This conversion is really a change of phase in the material, and the transformation usually results in increased strength and hardness, different physical properties, and very significant differences in response to electricity and other forms of activating energy. It seems possible that materials with greater resistance to cosmic and solar radiations will be developed. Perhaps even the ionizing Van Allen radiations, which cause so much disturbance in the electronic devices of flying space vehicles, may be soon overcome (G. C. Kennedy Lecture, University of California, Los Angeles, March 1964). Thus, ultrahigh pressure research holds the promise of developing materials with electronic properties far superior to those presently available. Moreover, the unfolding and rapidly deepening knowledge of high pressure physics will as a matter of natural consequence lead to the emergence of ultrahigh pressure and temperature techniques. These may bring with them the extension of solid-solid transitions to a number of tactically important high-strength materials - which would then have hitherto unknown mechanical properties.

While these hopes are enticing, it behooves us to examine on what foundations they have grown. In other words, what is the state of the art of ultrahigh pressure research? What is meant by "ultrahigh pressure", how is it produced, and what has to date been accomplished with it?

DEVELOPMENTS IN ULTRAHIGH PRESSURE

There are several ways of producing ultrahigh pressures. Working with shock tubes or with high explosives used as shaped charges, or yet, with plane-wave generators and driver plates, extremely high transient pressures can be achieved. The magnitude of the pressure that can be attained is function of the shock velocity and also of the inertia of the material exposed to shock. The greater the apathy of the material, the higher the dynamic pressure induced. A few random examples will illustrate the point. The highest pressure produced by shock in Cu is 9550 kilobars (1 kb = 1000 atm or roughly 15,000 lb/sq in.), in uranium it is 6450 kilobars, while in plexiglass it is only 2000 kilobars and, just as a matter of reference, gun pressure is around 100 kilobars (George E. Duvall, Lecture on Ultrahigh Pressures, University of California, March 1965).

The duration of a shock wave is about 2 microseconds with the most commonly used explosives, which are TNT, RDX, Composition B, Cyclotol 77/23 and, of course, PETN, and primacord.

It may be noted here that if the target of a high-explosive-induced shock wave is a near theoretically rigid wall, the shock wave is reflected. The pressure produced by the reflected shock wave is much higher than the pressure generated by the shock wave itself, and it can be higher by as much as a factor of 10.

One of the most noteworthy effects of shock on metals and alloys is hardening. Apparently even carbides are hardenable by shock, but techniques for doing it are still in the development stage. The greatest advance in the application of shock energy has been made in the domain of explosive forming.

Besides explosive hardening and forming, remarkable results have been achieved in explosive welding, especially in cladding a number of dissimilar materials into composites made up of several strata whose different properties combine into an astonishing synthesis - or symphony, if one prefers - of new physical and mechanical characteristics.

This is, in a cursory appraisal, the picture of ultrahigh pressure produced by explosives. The situation is an entirely different one with nondynamic or, putting it positively, static extremely high pressure.

First of all, static pressure is supposed to be of infinite duration, and producing the pressure requires mechanical equipment and pressure-transmitting and confining media. The transmitter can be a gas, a liquid, or a solid. When liquids are used, the pressure cannot be higher than about 35 kilobars, because most liquids freeze at 30 or 35 kilobars. For this reason there is apparently a tacit agreement to use the term "ultra-high pressure" (UHP) for the range above 30 kilobars. Another great difference between dynamic and static UPH is that the maximum static

pressure produced is approximately 500 kilobars, as opposed to the awesome dynamic pressure of 9550 kilobars, or, in round figures, 10,000 kilobars mentioned earlier.

The outstanding work in static ultrahigh pressure research is that of the late Professor Bridgman of Harvard University. Between 1909 and 1960 Bridgman wrote 199 papers dealing with "High Pressure Effects". His "Collected Experimental Papers" alone, published by Harvard University Press in 1964, fill seven respect-commanding volumes. There is hardly an investigator interested in the high pressure field who could forego studying the fundamentals set down by Bridgman. His principles of massive support for compression anvils and for pistons is still valid and used with some modification all over the world.

The Bridgman Anvils

Bridgman anvils are laboratory equipment consisting of two broad-angle chamfered, flat-faced compression dies made of a tough grade of tungsten carbide which is buttressed against lateral flow by tight-fitting steel rings. They are used in experiments with specimens of small volume and surface area. While they can withstand fairly high pressures (up to CCA 400 kilobars), the size of the sample is too small for comprehensive information in high temperature work (Figure 1).

Yet it is generally recognized that it is the Bridgman technique of "massive support" which initiated the development of two improvements enabling today's investigators to apply ultrahigh pressures to larger areas and greater volumes of material specimens.

The two improvements are the use of multiple binding rings around the compressing anvils, and the embedding of the specimen into a self-gasketing material.

Figure 2 is a somewhat more sophisticated application of the massive support principle. Two cylinders, one large and one small, are bored along the same axis in a block of steel subsequently heat treated and ground. The large piston ends in an obtuse angle truncated cone, which offers the area of massive support, while a plasticized collar acts as gasket and provides the radial restraining forces around the upper end of the unattached or floating high pressure piston. The pressure distribution (measured by Zeitlin) deserves our attention. The press supplies 170 tons to the truncated anvil which, by reason of area difference, translates to the high pressure piston 146 kilobars. The resultant lateral pressure on the generatrix of the small piston is 10.5 kilobars, while at the contact face of the specimen cell the pressure reads 103 kilobars, ending at the face of the closing anvil in 72 kilobars.

A simple version of a compression cell is presented in Figure 3. The massive support idea is the same as in Figure 1, except for the cylinder block which with the piston and anvil constitute a closed die type of

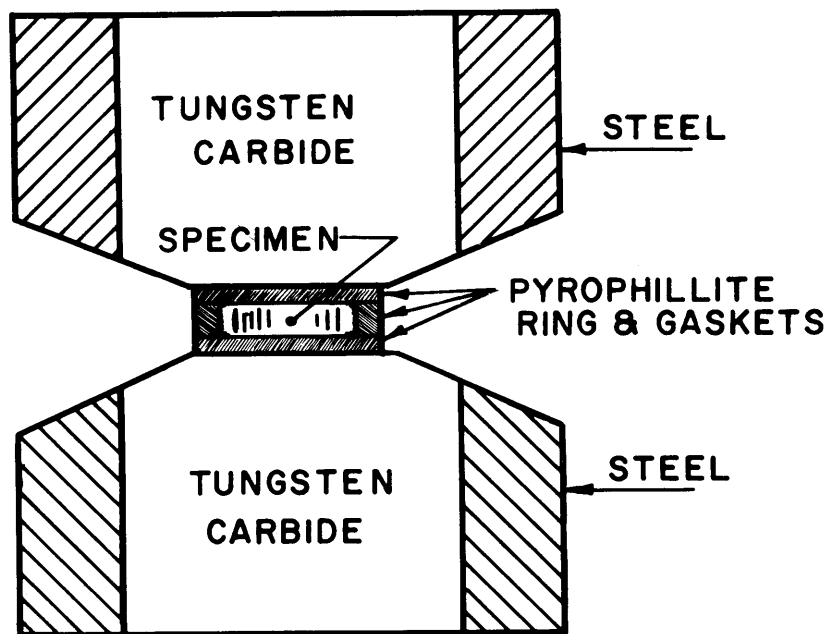


Figure 1. BRIDGMAN ANVILS

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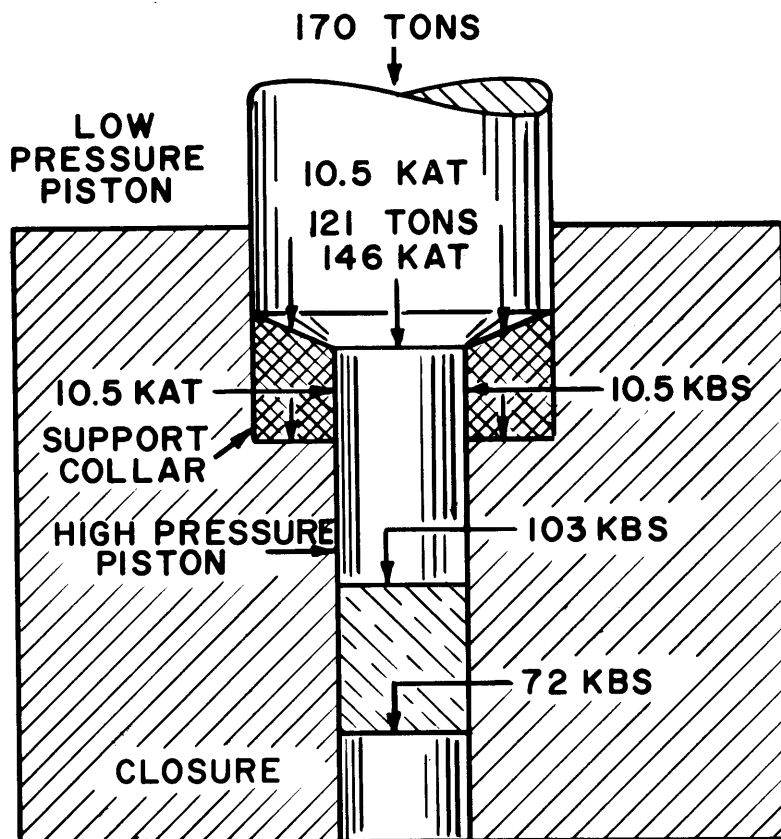


Figure 2. MASSIVE SUPPORT

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design. Lava is used in the form of a conical ring as a self-gasketing material.

In Figure 4 the laterally-buttressed massive support of Figure 2 evolves into a clear-cut and creatively designed lateral support principle. A steel band is the constraining chamber in which two opposed rams heading toward one another compress the pyrophyllite-embedded specimen. On both sides of the lateral support ring, a shaped steel ring is placed.

During pressing action the pyrophyllite will flow into and freeze solid in the rings, acting there as a selfsealing gasket. The three internal rings are surrounded by three concentric outer steel binding rings of which the inner one has a tapered OD, the central one is tapered both on the inside and on its OD, while the outer band has a matching taper on its ID.

Massive Support

What is assumed to be the ultimate in the application of the concept of massive support is represented in Figure 5. This has become known as the "belt apparatus". It was developed by Hall for General Electric.

In it, both the anvils and the constraining die benefit from the massive support principle. Two heavy rings surround each anvil. These binding rings are made of heat-treated steel, prestressed almost to their yield point by tapered interference fits, and girded by a soft, mild steel safety ring. The role of the soft ring is to prevent injury to personnel from fragments, should the binding rings fail under pressure.

The apparatus is water cooled, and can hold the specimen steadily at 2000 C under a pressure of 150 kilobars.

The specimen cell of the belt apparatus is quite complicated both in conception and execution. One of its prominent features is the sandwich gasket. In it one component relates to the next one with the logical concatenation of a stacked syllogism.

That the belt apparatus has merit is shown by the reproducibility of the sharp electrical resistance in bismuth, thallium, cesium, and barium. Measured in a Bridgman anvil these transitions, according to the old scale, were reported to occur at 24.9 kilobars for Bi, 44 for Tl, 54 for Cs, and 78 for Ba. A new scale was established with the belt apparatus. It reads 25.4 kilobars for Bi, 37 for Tl, 42 kilobars (instead of 54) for Cs, and 59 in the case Ba (instead of 78).

The tetrahedral arrangement shown in Figure 6 is a skeletal representation of four converging pistons. Their flat ends are chamfered as shown. The three lands of the chamfers at 109.47 degrees leave a flat area on each piston, forming an equilateral triangle. When the four pistons converge, their truncated cone ends leave an empty space in the shape

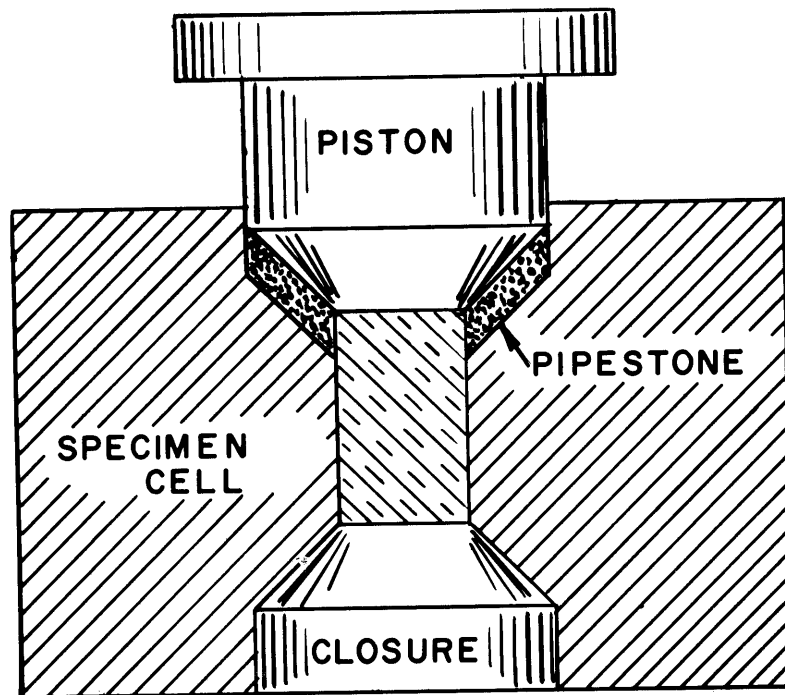


Figure 3. MASSIVE SUPPORT EXTENDED TO THE LOWER CLOSURE

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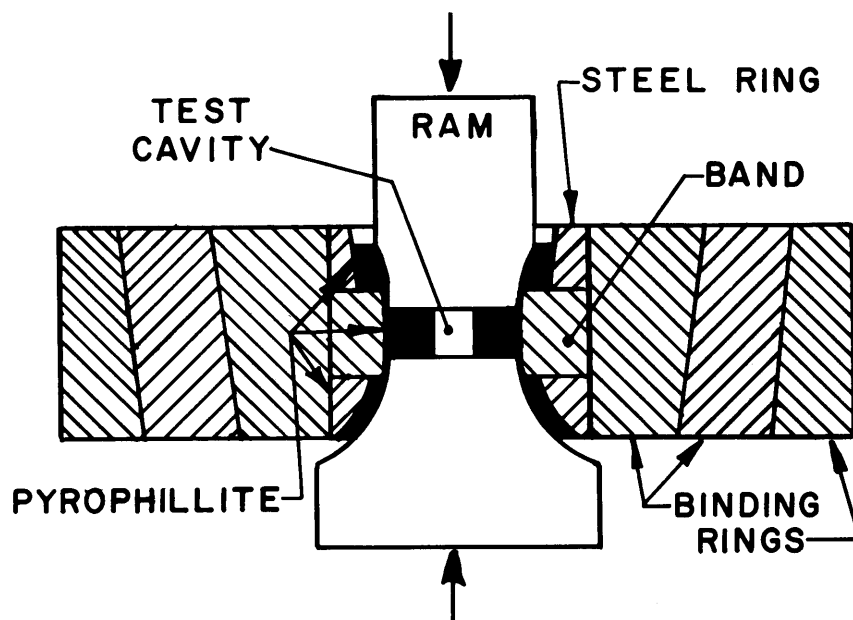


Figure 4. LATERAL SUPPORT PRINCIPLE

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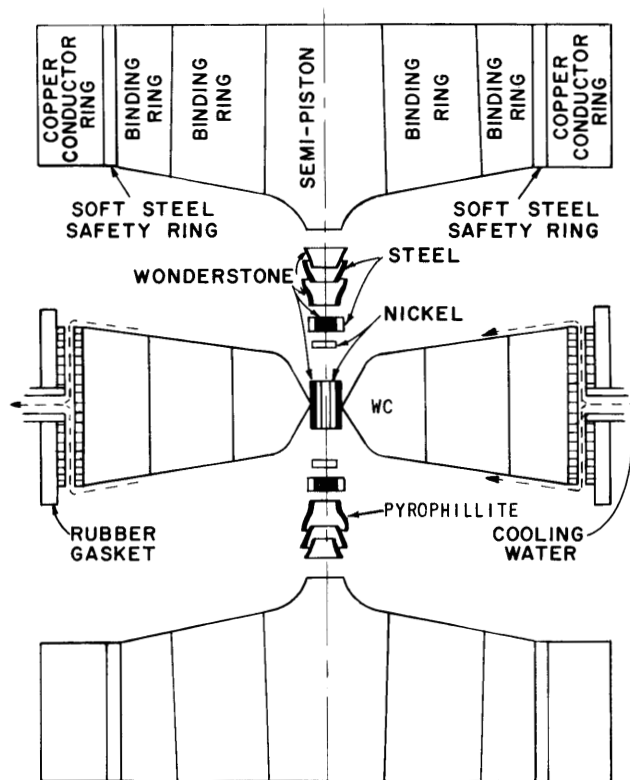


Figure 5. HALL'S BELT APPARATUS (G.E.)

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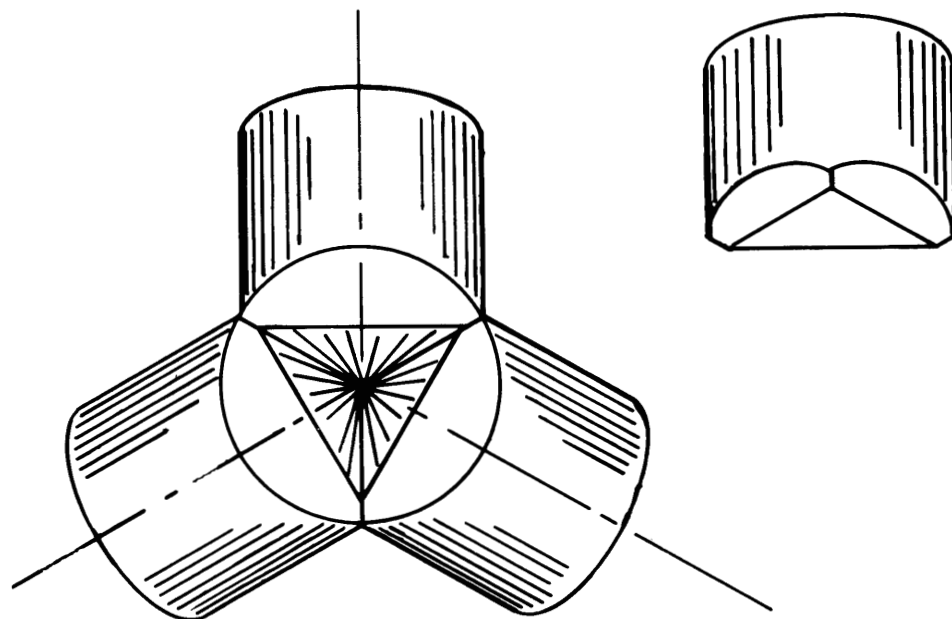


Figure 6. THE TETRAHEDRAL ARRANGEMENT

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of a tetrahedron. The piston ends are the anvils in this case. They are made of tungsten carbide.

The National Bureau of Standards designed a tetrahedral fixture as shown in Figure 7. Basically it is a hydraulic cylinder similar to a dashpot with a free floating piston in it. In the piston there is a tapered bore. Three of the tetrahedral anvils have their outer ends ground to conform to the tapered bore of the piston. The end of the fourth plunger is flat and butts against the upper closure of the cylinder when fluid under pressure forces the piston upward.

Figure 8 is a photograph of the fixture. On first sight such a device is attractive, but in usage the loss of force through friction posed severe problems. Teflon plugs in the anvil ends helped, yet as a whole it has been recognized that the limitations of the fixture are too great to replace effectively a tetrahedral press.

The Tetrahedral Press

With a regime pressure of 13,000 psi, the tetrahedral press represented in Figure 9 can produce a total pressure of 400 kilobars (400,000 atm) on the specimen. The design of the press is worth noting: simple flat links held in place by high strength pins join the three forged steel cylinder blocks. The function of the laterally mounted device is to take X-ray diffraction pictures of specimens under pressure.

The Hexahedral Arrangement

Figure 10 illustrates the principle of exerting UHP on a cube instead of a tetrahedron. Six converging pistons have their extremity so shaped as to leave, when all six meet, a cubic space for the compression of a specimen embedded in pyrophyllite.

A drawing of a hinged hexahedral apparatus is presented in Figure 11. The complexity of such a press and its higher cost weighed against its lower efficiency cause the tetrahedral press to be preferred. In favor of the hexahedral press is the very much easier preparation of specimens, as can readily be seen from an examination of Figure 12. Here the tetrahedral specimen cell is compared to the hexahedral one.

No. 1 - The specimen.

No. 2 and 4 respectively - The heating sleeve and two tabs surrounding the specimen.

No. 3 - Tetrahedron made of grade A lava or pyrophyllite (Tennessee Lava Corporation, Chattanooga, Tennessee).

No. 4 - Inner tab for heat transmission to sleeve No. 2.

No. 5 - Intermediate tab.

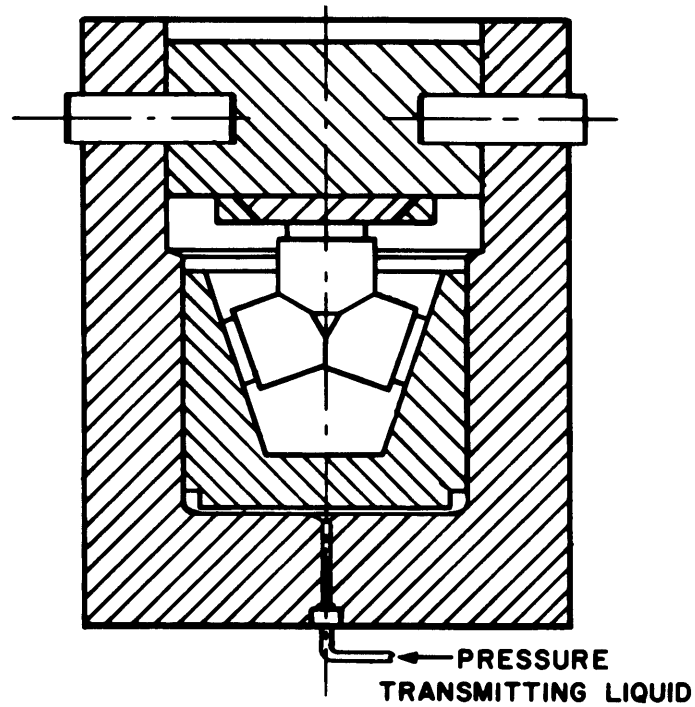
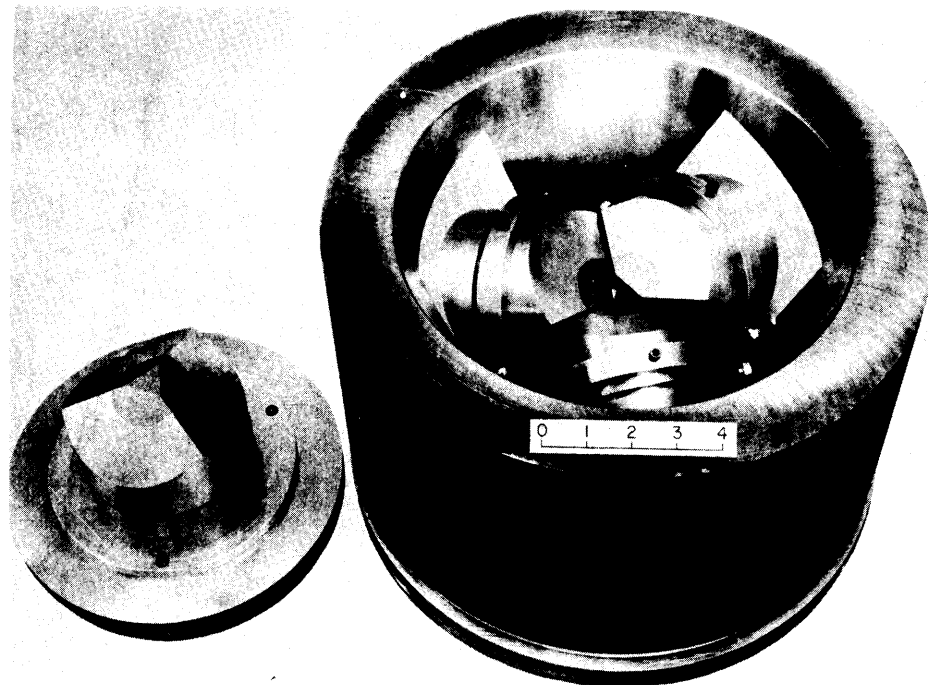


Figure 7. THE TETRAHEDRAL HYDRAULIC FIXTURE OF NATIONAL BUREAU OF STANDARDS
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Figure 8. THE TETRAHEDRAL FIXTURE OF NATIONAL BUREAU OF STANDARDS
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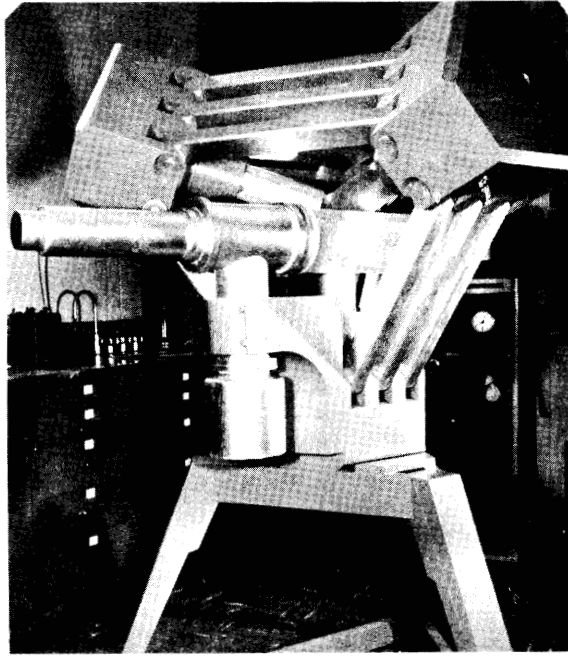


Figure 9. THE TETRAHEDRAL PRESS
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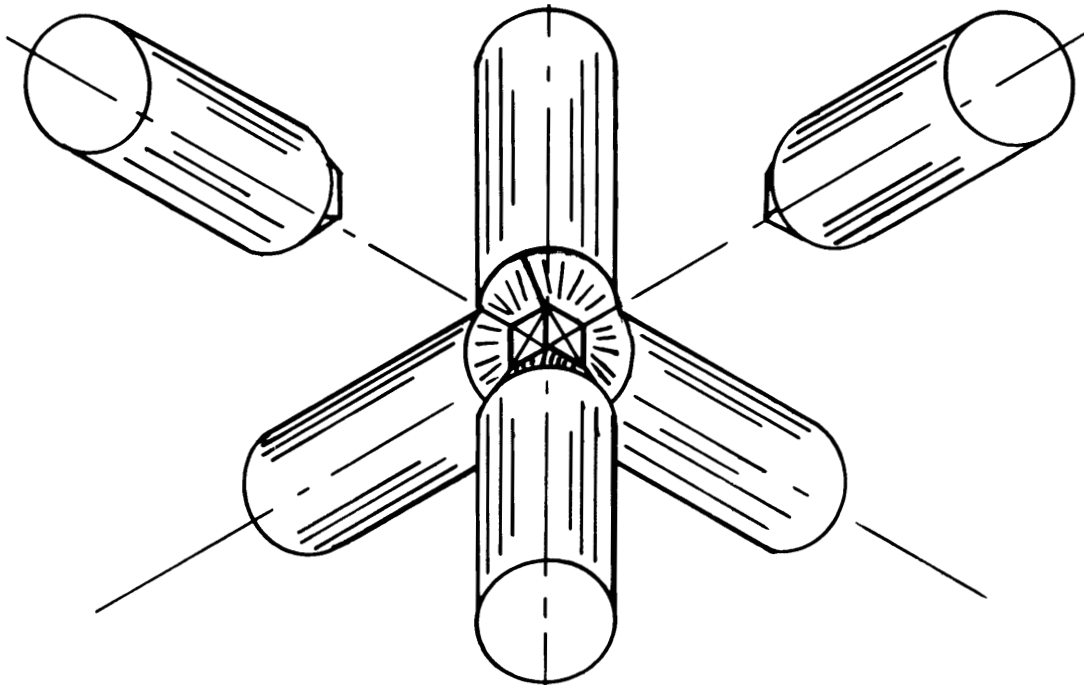
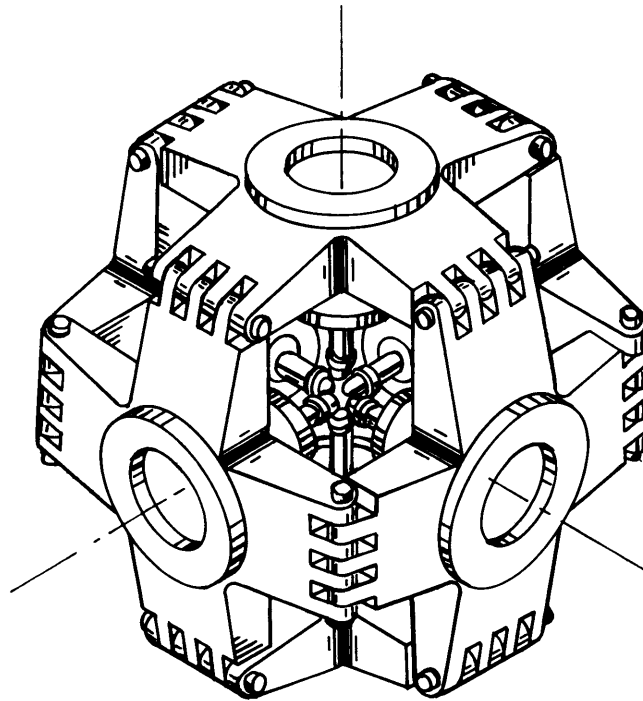


Figure 10. THE HEXAHEDRAL ARRANGEMENT
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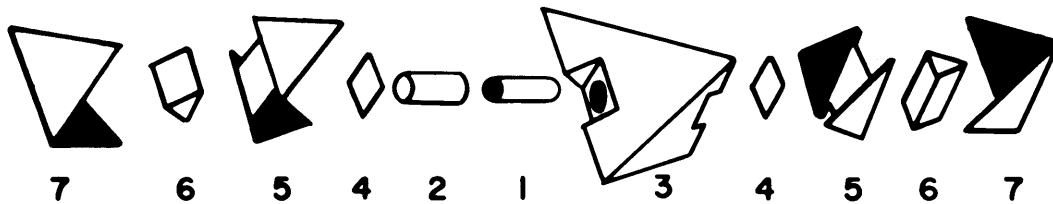


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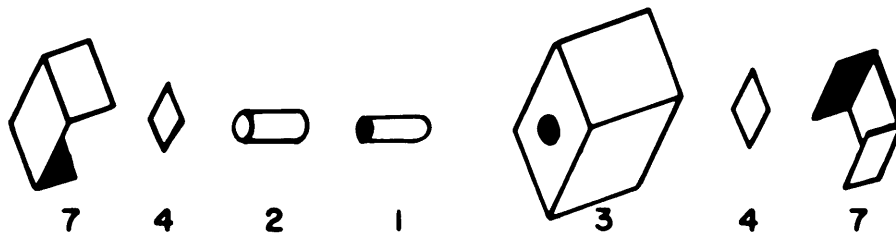
Figure 11. THE HINGED HEXAHEDRAL APPARATUS

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- | | | |
|--------------|---------------------|---------------------|
| 1. SPECIMEN | 2. HEATING SLEEVE | 3. CHARGE CONTAINER |
| 4. INNER TAB | 5. INTERMEDIATE TAB | 6. CAP |
| 7. OUTER TAB | | |



TETRAHEDRAL CONFIGURATION



CUBIC CONFIGURATION

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Figure 12. EXPLODED VIEW OF CAVITY ASSEMBLIES

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No. 6 - Cap, or pyrophyllite space filler between intermediate and outer tab.

No. 7 - Outer tab for contact with WC anvil.

The cubic sample holder shown at the bottom of Figure 12 is quite obviously much simpler, easier, and much less costly to machine and to provide with current transmitting metallic parts.

Heating Arrangement for a Pyrophyllite Tetrahedron Enclosed Specimen

There is an absolute minimum of two factors which must be known in ultrahigh pressure research - pressure and heat. To know the temperatures at which the experiment is carried out presents no difficulty. A thermocouple leading from the specimen to a classical galvanometer will be satisfactory. Pressure measurements require a more thought-demanding approach - calibration is necessary. For this, metals with known points of phase transition, indicated by a change of electrical resistance while under pressure, are used. Wires, 0.015- to 0.025-inch diameter, of such metals are placed into a small quantity of silver chloride and the whole is introduced into the tetrahedral cavity of the press. The silver chloride tetrahedron is prepared exactly as if it were a pyrophyllite specimen holder. The electrical current flowing through the metal wires will encounter sharp changes in resistance at the already known points of transition. These are the points that will serve as yardsticks of calibration. The change or changes in resistance will be indicated by an outside bridge circuit connected to a recorder (Figure 13).

An exposé (even a very brief one) which deals with ultrahigh pressures and their effects must evoke, in support of fundamental notions, Le Chatelier's law: "If a stress - heat or pressure or both - is applied to a system in equilibrium, the equilibrium is disturbed in a direction which tends to cancel the effect of the stress." It is a major aspect of Le Chatelier's principle that materials will generally respond to pressure by a tendency to contract, to become smaller in volume.

Assuming that we are dealing with a stable system, this contractive trend can be expressed by a relation based on the second law of thermodynamics, namely

$$\left(\frac{\partial V}{\partial P}\right)_T < 0.$$

Defining two commonly used terms for expressing the amount of contraction of a substance under pressure we have on the one hand:
compressibility

$$k = \frac{1}{V} \left(\frac{\partial V}{\partial P}\right)_T$$
$$\frac{\Delta V}{V_0} = \frac{V_0 - V}{V_0} = \frac{1}{V_0} \int_{P_0}^P V k dP;$$

and on the other hand compression where V is the volume at pressure P and V_0 is the volume at pressure P_0 . Compressibility indicates the rate of partial contraction of the material while compression gives the total volume alteration due to pressure.

It is interesting to note that compressibility is idiosyncratic in character; it varies from one substance to another. The variance is very great between gases and liquids and of lesser magnitude between liquids and solids. Accordingly, a volume change of 5 percent can be brought about in solid helium by a pressure of 50 bars (atmospheres), in cesium by 1400 bars (atmospheres), and in beryllium by as much as 100,000 bars or 100 kilobars.

The effect of extreme pressures on solids and more specifically on a single crystal has been under study of increasing intensity in the last four or five years. The number of university professors and other high caliber names in high pressure physics is too great to be enumerated here. A bibliography, courtesy of U. S. Bureau of Weapons and Defense Metals Information Center, is to be found in the appendix.

Single crystals are made up of atoms or combinations of atoms disposed in space in a three-dimensional periodic structure. The forces governing the spatial relationship between atoms depend on the kind of element we are dealing with. The atoms of crystals can be brought closer together by adequate pressure (see Figure 14). They can also be distorted, inducing a rearrangement of the atoms' electron shells (Figure 15). By extreme pressure nonmetallic atoms can be stripped of some of their electrons, changing the nonmetal into a metallic conductor. For example, iodine, phosphorus, and tellurium are materials which can be forced to act as metallic conductors. Over 100 kilobars of pressure is necessary to produce such changes.

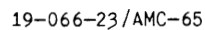
The changes produced in a periodic lattice when it is forced into another pattern is referred to as a polymorphic transition, and it is actually a new, different phase of the material. It has been observed that with each phase change goes a reduction of compressibility.

The presently producible pressures are not sufficient to bring about phase changes in all materials. Nor are all phase changes stable; most materials will not retain the new form they have assumed while under pressure. An outstanding example of nonreversing phase change is that of the mutation of graphite into the purest form of carbon - diamond.

Quite incidentally, diamonds from graphite are now produced for industrial applications. Their size is usually about one-half millimeter sides (0.020 inch). The largest diamond ever produced from graphite measures about 4 carats. By direct association, diamond leads us to carbon and thence to the effect of UHP on the iron-carbon diagram (Figures 16 and 17). Pressure lowers the eutectic temperature and the eutectic gets shifted to a lower carbon content. Most materials will also have their melting points affected one way or another by UHP. Figure 18 is an example of the influence of UHP on the melting point of some elements.

-
- FAR SIDE ANVIL
(NOT SHOWN)
- 1
- 2
- 3
- 4
- 5

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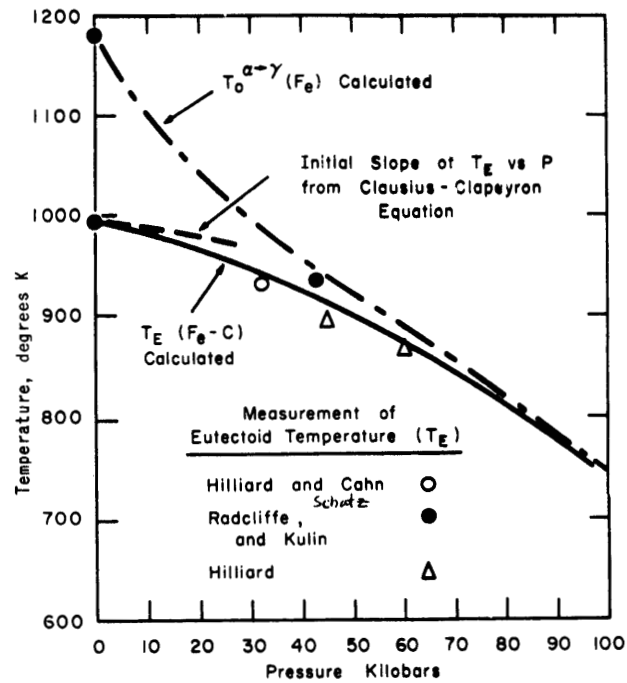


Figure 17. EFFECT OF PRESSURE ON THE EUTECTOID TEMPERATURE IN THE IRON-CARBON SYSTEM

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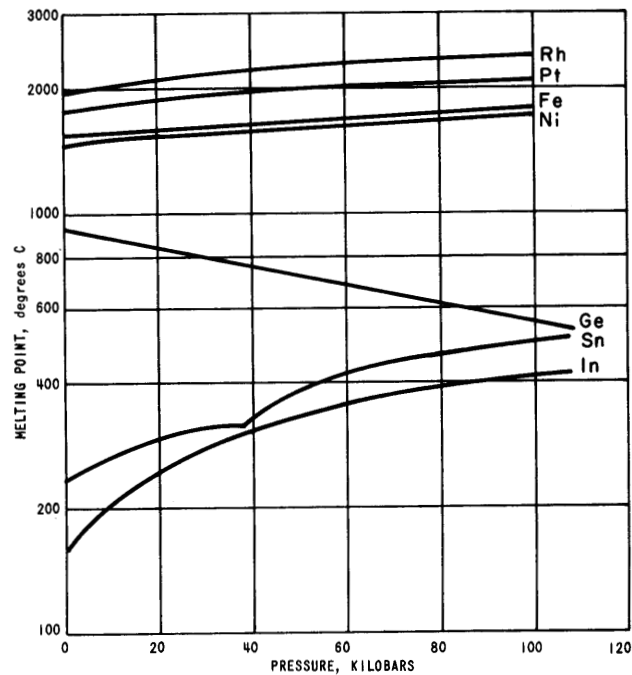


Figure 18. MELTING CURVES OF VARIOUS MATERIALS

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Returning to the most vital effect of UHP, the phase change, the stability or irreversibility of a transition appears to depend very much on the character, magnitude, and distribution of the cohesive forces that hold the atoms in their respective positions in their lattice. These cohesive forces constitute the internal pressure of the material. When a material is subjected to unusually high pressure, its atoms are forced toward one another: the material is compressed and, because the atoms repel each other when they come closer together, the internal pressure decreases. At first look, the opposite effect would seem to be true; namely, that on application of external pressure the internal pressure would increase. However, it must be borne in mind that it is the internal pressure that holds the material in its solid condition, while the external pressure tends to destroy it precisely by weakening, that is to say, by actually decreasing the internal pressure of the material.

In a crystal the internal pressure can be negative, but the sum of internal and external pressures must be positive and of great magnitude, or no stable system could be had. At ultrahigh pressure the internal and external pressures may approach equal values as is shown by the fact that compressibility decreases with additional pressure.

If a phase change comes about, the magnitude of the internal pressure will indicate whether the new phase will be irreversible. The greater the internal pressure, the better the prospect of a stable new phase or a new material. Conversely, a negative internal pressure will have for concomitant the return of the material to its original condition as soon as the externally applied pressure is taken off.

A telling comparison between materials of high and low internal pressure is the one made between carbon and bismuth (Figure 19). Bismuth has a very low, in fact, a negative internal pressure, and reverses to its original condition when the pressure that causes the phase change is released. Carbon on the other hand changes to diamond, and diamond remains stable; we say it is irreversible at atmospheric pressure.

THE SITUATION AS OF NOW

Great progress has been made in the last five years in the development of pressure-producing equipment, of interdisciplinary studies, of measurement techniques, methods and areas of investigation.

In equipment, machinery for pressures up to about 30 kilobars is relatively easily obtained, or available within short periods of delivery. When it comes to higher pressure, special presses have to be built and delivery will take a year or more. The attainable static pressure cannot as of now exceed 500 kilobars, and an immediate breakthrough that would raise this value by several factors is not hoped for.

As to interdisciplinary work, the Universities of Washington State, of Illinois, and principally of California (Los Angeles) have been conducting intensive research in UHP correlated with other disciplines such as geology, dynamic UHP and ultrahigh pressure chemistry. In this latter field new reactions were induced and new compounds were achieved.

It is in the area of measurement techniques that the most interesting and reward-promising achievements have been made. Differential thermal analysis is one of them. Others are techniques for low temperature measurement, infrared spectroscopy, X-ray diffraction, optical observation, and electron and nuclear resonance. Some investigators are now working on improved techniques of differential thermal analysis, on the application of X-ray diffraction at high temperatures, on methods for ferromagnetic resonance measurements, and the determination of index of refraction at UHP.

Anvil materials capable of withstanding higher pressures than 500 kilobars are also being sought, together with better anvil concepts. Perhaps better anvils could be made by shock hardening some of the tougher grades of tungsten carbide, or by surrounding the best available anvil materials with a constraining high pressure environment which would balance or even exceed the UHP of the specimen cell.

Although much remains to be done in UHP research and the challenge is great, the rewards are equally great and intensely stimulating. One of the most exciting results of the new techniques is what happened in experiments with boron nitride. As it normally occurs, boron nitride has a structure similar to that of graphite, except that the lattice is composed of boron and nitrogen atoms. It further resembles graphite by its low coefficient of friction, its slipperiness. Some people actually call it "white graphite". R. H. Wentorff of General Electric, proceeding on a hunch, applied ultrahigh pressure and high temperature to this intriguing material. It was a rewarding experiment. It had for spectacular result an entirely new material which Dr. Wentorff and associates promptly named "Borazon". It is a man-made material in the hardness range of diamond, in fact, the only material capable of scratching diamond. As Figure 20 shows, no other mineral is as hard as diamond. Borazon (not shown) is the only one that reaches up to the top of Mohs' scale. Another valuable quality of Borazon is that it is more oxidation-resistant than diamond.

In conclusion, it seems warranted to state that the general result of each particular research endeavor in this new field will be better equipment, better methods of investigation via newly developed measurement techniques, and, ultimately, a growing number of improved old materials as well as new materials which will have hitherto unachieved properties and capabilities. Clearly, in the not-too-distant future, UHP research will make valuable contributions to both science and technology.

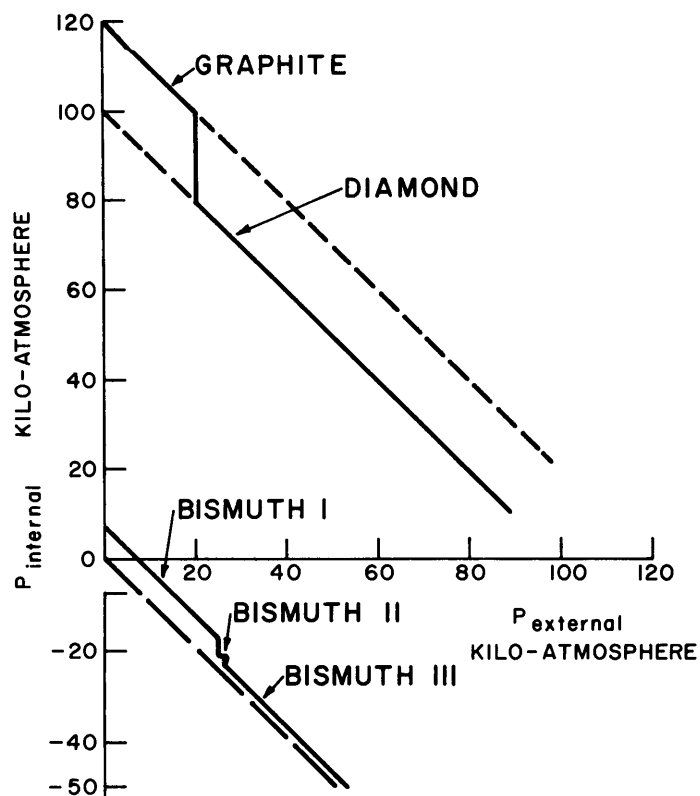


Figure 19. INTERNAL PRESSURE OF DIAMOND AND BISMUTH
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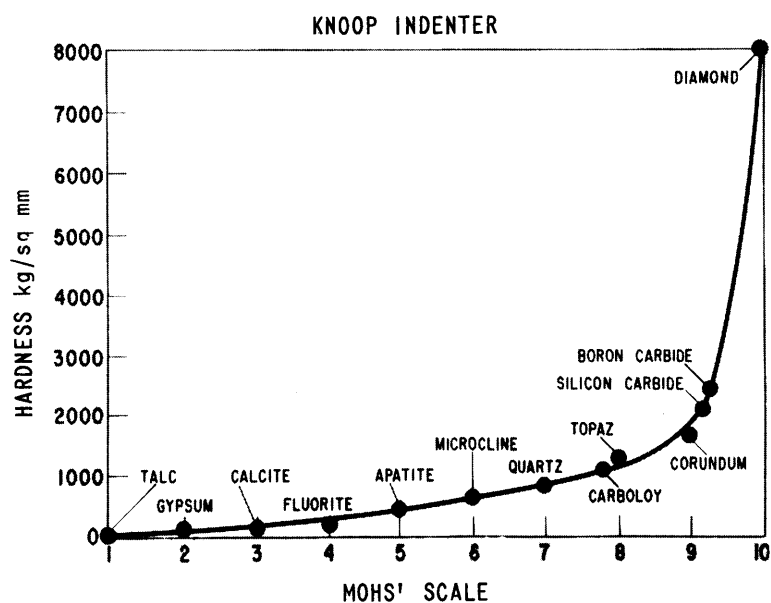


Figure 20. HARDNESS OF MINERALS VERSUS HARDNESS OF DIAMOND
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APPENDIX

EFFECTS OF PRESSURE ON PROPERTIES OF METALS*

At the request of the Bureau of Weapons, the Defense Metals Information Center collected the attached information for the Committee on "Metalworking Processes and Equipment Program".

The material enclosed consists of:

(1) A bibliography of pertinent articles dealing with the effects of high pressure on metals, equipment for processing materials in high-pressure environments, and precautions for instrumentation of high-pressure investigations.

(2) Abstracts of some of the articles available at Battelle are included in the bibliography. These abstracts convey the opinions of the authors or describe the contents of the articles. For these reasons, they should be considered as unevaluated.

Part I. Selected References on Effects of Pressure on Properties of Metals

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Part II. Abstracts of Some Articles Listed in the Bibliography

- 1c. BUNDY, F. P.
 Absolute pressure thermal emf's were measured for constantan, Pt, Ni, alumel, Pt-10%Rh, Cu, chromel, and Ni-18%Mo for a ΔT of 100 C over a pressure range of 0 to 100,000 atm. Corrections due to pressure for common thermocouples made of pairs of three metals were deduced. A number of thermocouple pairs have been compared at temperatures up to 1200 C and pressures up to 75,000 atm.
- 1f. HILLIARD, J. E., and CAHN, J. W.
 General discussion on the effect that pressure can be expected to exert on various factors known to influence the rate of solid-state transformations. Use of activation volume to describe pressure dependence is briefly discussed together with graphical construction for estimating pressure displacement of phase boundaries in binary systems.

- 1g. KAUFMANN, L., LEYENAR, A., and HARVEY, J. S.

The effect of hydrostatic pressure on the thermodynamic and kinetic characteristics of fcc <---> bcc reactions in iron base alloys has been studied. It was found that the stability of the fcc phase is increased by hydrostatic pressure as predicted by theory. Moreover, appropriate pressure-temperature cycling can be utilized to suppress the potency of bcc martensitic nuclei and thereby lead to changes in the transformation characteristics at atmospheric pressure.

5. BASS, J., and LAZARUS, P.

There is no effect of pressure on the mobility of the carbon atoms.

8. BERESENEV, B. I., VERESHCHAGIN, L. F., and RYABININ, YU. N.

Presentation of the work done in the Soviet Union on the extrusion of metals by high-pressure liquids.

Description of the design of preliminary laboratory device for drawing metals into wire and rolling metals with free-running rollers, both under high-pressure liquids is given. Also included are design details of a laboratory apparatus developed for cold drawing and rolling, currently of aluminum specimens, with freely rotating rollers in liquid under pressures of about 10,000 kg/cm² (142,000 psi).

10. BERESENEV, B. I., VERESHCHAGIN, L. F., and RYABININ, YU. N.

Work performed in the extrusion of aluminum with various fluids showed that the magnitude of the pressure needed for the flow and also that the surface quality of the deformed metal depends on the fluid. Methods for reducing the pressure needed for extrusion have been evaluated.

27. DAVIDSON, T. E., and HOMAN, C. G.

The observed structural changes consisted of deformation localized along grain boundaries, which appear to be a boundary migration phenomena, and widespread slip and cross slip. Subsequent deformation is progressive with increased pressure and is substantially different in type from the deformation characteristics of uniaxial compression.

28. DAVIS, R. S.

Study of the polymorphic phase transformation in iron at high pressures. Data obtained from experiments with static high pressure apparatus and dynamic shock high pressure technique are compared. Evidence for a possible new phase in iron is summarized.

29. DeVRIES, K. L., BAKER, G. S., and GIBBS, P.

Summary of all "known" high pressure work done on solid since 1947. Data are presented in the form of 40 tables and more than 500 figures. Some 250 references are summarized with descriptions given on apparatus, procedure, and results.

31. FIORENTINO, R. J., SABROFF, A. M., and BOULGER, F. W.

Hydrostatic extrusion of AISI 4340 at an extrusion ratio of 2:1 was achieved at a pressure roughly 25 percent less than that required by conventional extrusion. Commercial-purity aluminum was extruded hydrostatically at extrusion ratios of up to 20:1 at pressures about 15 percent less than those required by conventional techniques.

43. JAMIESON, J. C.

Brief review of previous studies on crystalline structures at high pressures. X-ray analyses show that titanium and zirconium metal have a distorted bcc structure. This phase persists on pressure release. The normal hexagonal close-packed structures are recovered when the metals are heated. Hafnium metal shows no such transition.

45. KAUFMAN, L., CLOUGHERTY, E. V., and WEISS, R. J.

Determination of the temperature of the bcc <---> fcc transition as a function of pressure from measurements of the electrical resistance of iron over a temperature range of 325 to 1425 K at pressures up to 95 Kb. The results are used in conjunction with available evidence of the thermodynamic, volumetric, and physical properties of bcc and fcc iron to evaluate the factors which affect the relative stability of these irons as a function of temperature and pressure.

46. KAUFMAN, L., ET AL

The experimental investigations on the effect of high hydrostatic pressure on the phase transformations in various substitutional iron-base alloys, including iron-chromium, iron-nickel, and iron-silicon, yield data which are in close agreement with theoretical prediction. A study on the effects of pressure on iron-carbon alloys shows a general shift of the equilibrium phase boundaries to lower carbon contents and temperatures with pressure. Pressure also acts to retard both the act of tempering and the isothermal transformation of metastable austenite.

49. KRUPNIKOV, K. K., BAKANOVA, A. A., BRAZHNIK, M. I., and TRUNIN, P. F.

Presentation of the results of an investigation of the impact compressibility of titanium, molybdenum, and tantalum over a pressure range of 1 to 5 million atm, and of iron at a pressure of 9 million atm. The determined adiabatic lines of impact and the zero isotherms of each metal are presented.

50. KULIN, S. A., ET AL.

The kinetics of recrystallization of polycrystalline copper (99,999% purity) cold-rolled to 98% reduction have been determined for the temperature range 80-170 C at atmospheric pressure and at 42 kilobars. High pressure is found to retard both the initiation and rate of recrystallization. The results of a series of experiments designed to investigate pressure-quenching in several different iron-nickel alloys are reported. Thermodynamic data obtained at one atmosphere is used to correlate the high-pressure transitions in thallium and tin. Several iron-carbon alloys and plain carbon steels ranging from 0.08 to 1.23 weight percent carbon content were subjected to various heat treatments at a pressure of 42 kilobars. A series of experiments in which pressure is used to enhance the mechanical properties of selected steels is described. The effective stress-strain curves for both a one-atmosphere and a 37-kilobar heat-treated 4320 steel specimen are shown. A definite increase in strength is seen in the pressure-treated steel. Extrapolating the data to obtain approximate yield strengths results in values of 150 ksi and 200 ksi, respectively.

53. MELNIKOV, L. A., SOKOLOV, B. K., and STREGULIN, A. I.

Effect of compressive load exerted on an Fe-Ni alloy specimen in a special pressure chamber on the transformation of martensite into austenite at 360 to 520 C.

54. MURRELL, S. A. F.

Consideration of the effects of high pressure on the fracture of ductile materials and on the ductile-brittle transition.

59. PRESSURE TECHNOLOGY CORPORATION OF AMERICA

Correlative fluid-extrusions were performed on sintered polycrystalline tungsten and beryllium, and on a single crystal of beryllium. There were indications that crack-free fluid-to-fluid extrusions should be possible for all materials, with additional modes of deformation apparently activated at higher pressure levels.

Analysis is presented on the role of pressure in plastic deformation, with extrusion to high-energy forming.

62. PUGH, H. L. D., and GREEN, D.

The results of tests to determine the critical thickness of mild steel sheet of a given area which will just withstand various hydrostatic pressures are given. Tensile tests under pressure have been carried out on copper, aluminum, zinc, Mazak, and cast iron. Some of these results are: (1) zinc and Mazak showed an abrupt change from a very brittle to a very ductile behavior over a very narrow range of pressure, and (2) although all metals tested showed increased ductility with pressure, the relation was not linear.

63. RADCLIFFE, S. V., SCHATZ, M., and KULIN, S. A.

The effect of a pressure of 42 kb both on phase equilibria at temperatures up to 1100 C and on the kinetics of the isothermal transformation of austenite at temperatures below the eutectoid were investigated for high-purity Fe-C alloys containing 0.08 to 1.23 weight % C. At this pressure, the austenite ---> carbide and austenite ---> ferrite phase boundaries are displaced toward lower C contents. The eutectoid temperature is lowered from 723 to 660 ± 5 C and the eutectoid composition from 0.80 to approximately 0.25 weight percent C. The acicular bainitic structures normally characteristic of decomposition below 450 C are replaced at 42 kb by irregular ferrite-carbide aggregates and by direct precipitation of carbide from austenite.

64. Research on Strengthening Mechanisms Produced by High Dynamic Pressures in Iron Base Alloys.

Fundamental research shall be performed to establish the mechanisms of the strengthening effects produced by high dynamic pressures in selected iron-base alloys. The high dynamic pressures are generated by explosives. The initial effort will be concerned with inducing various behaviors in selected iron-base alloys by exposure to shock pressures of different magnitudes and determining the mechanical properties. The major emphasis will be placed on a detailed investigation of the mechanisms responsible for the enhancement of the strength properties.

67. RYABININ, YU. N., ET AL.

Data on duralumin, Fe, and stainless steel at pressures from 1 to 32,000 kg/cm².

69. SCHWARTZ, C. M., and WILSON, W. B.

Application of ultrahigh pressure (greater than 1.5 million psi) to metallurgical research. Effect of pressure on structural, phase, and density transformations in solid are reported.

71. SILVERMAN, S. M., GODFREY, LOREN, HAUSER, H. A., and SEAWARD, E. T.

The effect of high dynamic pressures generated by strong shock waves on the metallurgical properties of selected iron-base alloys was investigated. The effect of shock wave duration, repeated shocks on a single test specimen, increasing shock wave intensity, and post shock heat treatment on the yield and tensile strength of H-11 tool steel and 25% nickel steel were studied. As a result of explosive shock hardening, H-11 steel increased in yield strength from 235 ksi in the preshocked condition to 340 ksi in the as-shocked (360 kilobars) condition, while the 25% nickel steel showed yield strength increases from 235 ksi in the austenitized-plus-aged condition to 255 ksi in the shocked-plus-aged condition. These increases in yield strength were brought about without any significant macroscopic plastic deformation.

73. SMITH, R. C.

Low-carbon steel is subjected to initial dynamic compressive or tensile loads with variations of the load intensity for study of the elastic strain. The specimens are then aged at 68-250 F and retested. Upper and lower yield stresses and inelastic microstrain are used to evaluate the extent of damage and recovery with correlations applied for predicted yield phenomena.